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UTILITY PATENT APPLICATION TRANSMITTAL
(Large Entity)

(Only for new nonprovisional applications under 37 CFR 1.53(b))

Docket No.
15851-1
Total Pages in this Submission

06/30/00 JC870 U.S. PTO

TO THE ASSISTANT COMMISSIONER FOR PATENTS
Box Patent Application
Washington, D.C. 20231

Transmitted herewith for filing under 35 U.S.C. 111(a) and 37 C.F.R. 1.53(b) is a new utility patent application for an invention entitled:

PROCESS FOR COATING SUBSTRATES WITH CATALYTIC MATERIALS

JC836 U.S. PTO 09/607211 06/30/00

and invented by:

BILLY T. UPCHURCH, DAVID R. SCHRYER and ERIK J. KIELIN

If a CONTINUATION APPLICATION, *check appropriate box and supply the requisite information:*

☐ **Continuation** ☐ **Divisional** ☐ **Continuation-in-part (CIP)** of prior application No.: ______

Which is a:

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Which is a:

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Enclosed are:

Application Elements

1. ☒ Filing fee as calculated and transmitted as described below
2. ☒ Specification having 11 pages and including the following:
   - a. ☒ Descriptive Title of the Invention
   - b. ☒ Cross References to Related Applications *(if applicable)*
   - c. ☒ Statement Regarding Federally-sponsored Research/Development *(if applicable)*
   - d. ☐ Reference to Microfiche Appendix *(if applicable)*
   - e. ☒ Background of the Invention
   - f. ☒ Brief Summary of the Invention
   - g. ☒ Brief Description of the Drawings *(if drawings filed)*
   - h. ☒ Detailed Description
   - i. ☒ Claim(s) as Classified Below
   - j. ☒ Abstract of the Disclosure

23351
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P01ULRG/REV04

UTILITY PATENT APPLICATION TRANSMITTAL
(Large Entity)

(Only for new nonprovisional applications under 37 CFR 1.53(b))

Docket No.
15851-1
Total Pages in this Submission

Application Elements (Continued)

3. ☒ Drawing(s) *(when necessary as prescribed by 35 USC 113)*
   - a. ☒ Formal Number of Sheets **ONE**
   - b. ☐ Informal Number of Sheets ______
4. ☒ Oath or Declaration
   - a. ☐ Newly executed *(original or copy)* ☒ Unexecuted
   - b. ☐ Copy from a prior application (37 CFR 1.63(d)) *(for continuation/divisional application only)*
   - c. ☒ With Power of Attorney ☐ Without Power of Attorney
   - d. ☐ *DELETION OF INVENTOR(S)*
     Signed statement attached deleting inventor(s) named in the prior application, see 37 C.F.R. 1.63(d)(2) and 1.33(b).
5. ☐ Incorporation By Reference *(usable if Box 4b is checked)*
   The entire disclosure of the prior application, from which a copy of the oath or declaration is supplied under Box 4b, is considered as being part of the disclosure of the accompanying application and is hereby incorporated by reference therein.
6. ☐ Computer Program in Microfiche *(Appendix)*
7. ☐ Nucleotide and/or Amino Acid Sequence Submission *(if applicable, all must be included)*
   - a. ☐ Paper Copy
   - b. ☐ Computer Readable Copy *(identical to computer copy)*
   - c. ☐ Statement Verifying Identical Paper and Computer Readable Copy

Accompanying Application Parts

8. ☐ Assignment Papers *(cover sheet & document(s))*
9. ☐ 37 CFR 3.73(B) Statement *(when there is an assignee)*
10. ☐ English Translation Document *(if applicable)*
11. ☐ Information Disclosure Statement/PTO-1449 ☐ Copies of IDS Citations
12. ☐ Preliminary Amendment
13. ☒ Acknowledgment postcard
14. ☒ Certificate of Mailing
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UTILITY PATENT APPLICATION TRANSMITTAL (Large Entity) (Only for new nonprovisional applications under 37 CFR 1.53(b))	Docket No. 15851-1
	Total Pages in this Submission

Accompanying Application Parts (Continued)

15. ☐ Certified Copy of Priority Document(s) *(if foreign priority is claimed)*

16. ☐ Additional Enclosures *(please identify below)*:

Fee Calculation and Transmittal

CLAIMS AS FILED					
For	#Filed	#Allowed	#Extra	Rate	Fee
Total Claims	24	- 20 =	4	x $18.00	$72.00
Indep. Claims	1	- 3 =	0	x $78.00	$0.00
Multiple Dependent Claims (check if applicable) ☐					$0.00
				BASIC FEE	$690.00
OTHER FEE *(specify purpose)*					$0.00
				TOTAL FILING FEE	$762.00

☐ A check in the amount of to cover the filing fee is enclosed.

☒ The Commissioner is hereby authorized to charge and credit Deposit Account No. 14-0116 as described below. A duplicate copy of this sheet is enclosed.

- ☒ Charge the amount of $762.00 as filing fee.
- ☒ Credit any overpayment.
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- ☐ Charge the issue fee set in 37 C.F.R. 1.18 at the mailing of the Notice of Allowance, pursuant to 37 C.F.R. 1.311(b).

Signature

HILLARY W. HAWKINS
REG. NO. 42,235

Dated: 6-30-00

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cc:

07/03/00

CERTIFICATE OF MAILING BY "EXPRESS MAIL" (37 CFR 1.10) Applicant(s): BILLY T. UPCHURCH ET. AL.	Docket No. 15851-1

Serial No.	Filing Date	Examiner	Group Art Unit

Invention: PROCESS FOR COATING SUBSTRATES WITH CATALYTIC MATERIALS

06/30/00 JC80 U.S. PTO

I hereby certify that the following correspondence:

NEW UTILITY PATENT APPLICATION

(Identify type of correspondence)

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ATTACHMENTS
- return postal card
- org & 1 cy transmittal - 3 pgs
- specification, claims, abstract - 11 pgs
- declaration (NASA Form 1538) - 2 pgs
- fee - $762
- 1 sheet FORMAL drawing

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P06A/REV02

EK456804778

PROCESS FOR COATING SUBSTRATES WITH CATALYTIC MATERIALS

Claim of Benefit of Provisional Application

Pursuant to 35 U.S.C. §119, the benefit of priority from provisional application 60/141,729, with a filing date of June 30, 1999, is claimed for this non-provisional application.

Origin of the Invention

The invention described herein was jointly made by employees of the United States Government and contract employees during the performance of work under a NASA contract which is subject to the provisions of 35 USC 202 in which the contractor has elected not to retain title.

Background of the Invention

1. Field of the Invention

This invention relates generally to the process of coating substrates with one or more catalytic components to form a catalyst. It relates particularly to the process of layering one or more catalytic components onto a honeycomb monolith to form a carbon monoxide oxidation catalyst which combines CO and O_2 to form CO_2, or alternatively, a volatile organic compound oxidation catalyst which combines the compound and O_2 to form CO_2 and H_2O.

2. Description of the Related Art

The catalytic conversion of carbon monoxide to carbon dioxide in the presence of oxygen is useful to a number of fields. Applications in which CO oxidation catalysts

may be successfully employed include the following:(i) catalytic removal of CO in air-purification systems, especially for enclosed spaces; (ii) removal of CO in filter canisters, and the like, for personal breathing apparatuses; (iii) removal of CO from combustion products of cigarettes; (iv) removal of CO from exhaust gases expelled from gasoline- and diesel-powered internal combustion engines; and (v) catalytic conversion of dissociation products in CO_2 lasers to maximize laser power and life, and minimize laser weight, size, and engineering complexity. Each of these and other applications require a different embodiment of a CO oxidation catalyst and place a different emphasis on one or another quality. Thus, a catalyst for an air-purification system necessarily must have a high throughput, while a catalyst for an internal combustion engine requires the capacity to operate over a broad temperature range, and cost per unit takes on greater significance in the cigarette application. Improvements in CO oxidation catalysts are continually being sought to increase the versatility, effectiveness, durability, activity, and operating life of the CO oxidation catalyst.

Several patents, e.g., U.S. Pat. Nos. 4,912,082 and 4,991,181 to Upchurch and U.S. Pat. Nos. 4,818,745 and 4,808,394 to Kolts, disclose compositions useful to CO_2 laser applications. During the operation of a CO_2 laser, CO_2 decomposes into CO and O_2 in the laser's electrical discharge zone. The concentration of the dissociation products increases throughout the laser's operation, while the concentration of CO_2 correspondingly decreases. Both the loss of CO_2 and the build-up of O_2, which scavenges electrons from the CO_2 molecules, significantly reduce the lazing power and degrade the performance of the laser. This problem may be addressed either by continually replacing the dissociation products with fresh CO_2 during the laser's operation or by using an ambient temperature CO-oxidation catalyst. The former is expensive and, especially for most airborne and space applications, unworkable because of the weight penalty. Hence, the latter is preferred, but the catalyst must have an extended activity life for most applications.

In U.S. Pat. Nos. 4,994,247 to Tooley and 5,017,357 to Kolts, CO-oxidation catalyst compositions are disclosed which are suitable for a number of applications

including the minimization of CO in tobacco smoke; removal of CO for personal breathing masks, e.g., those worn by miners; and CO_2 laser applications. Matsuyama, in U.S. Pat No. 4,117,082, and Harrison, in U.S. Pat. No. 5,051,393, disclose CO-oxidation catalyst compositions developed for use in minimizing carbon monoxide and/or unburnt hydrocarbons from vehicle exhaust. U.S. Pat No. 4,639,432 to Holt discloses CO-oxidation catalyst compositions directed towards the previously stated problems and also towards air-purification or ventilation systems for the removal of CO from confined spaces, especially where traditional ventilation methods are difficult or unfeasible. Examples include nuclear submarines and areas around welding equipment.

In many applications it is also highly desirable, if not necessary, to remove hydrocarbons and other volatile organic compounds from the air via oxidation to CO_2 and H_2O without the aid of filters and with minimal heating of the catalyst. By way of example, there has been a long-standing need for a method to remove volatile organic compounds from indoor air-- i.e., breathable air in enclosed spaces such as homes, automobiles, airplanes, ships, boats, and industrial plants where there may be high concentrations of said compounds. Other significant long-standing needs include the need to purify compressed air and other oxygen-containing gases, as well as the employment of personal safety masks in the removal of volatile organic compounds from the atmosphere. There has also been a need for such a method in selective chemical sensors and catalytic converters for combustion processes, including internal combustion engines which utilize gasoline, diesel, natural gas, and alcohol fuels.

Considering the range of applications and requirements specific to each, there is an ever present need to develop new, effective oxidation catalyst compositions and/or improved processes for preparing effective oxidation catalyst compositions. Any improvement which increases the versatility, effectiveness, durability, activity, and/or operating life of the catalyst or the process for making such, satisfies this need.

Supported catalysts - specifically, supported carbon monoxide oxidation catalysts - may be prepared by (i) coating a support with "catalytic paint"; (ii) impregnation with precipitation agents in one or multiple steps; (iii) impregnation

followed by calcination or firing; and (iv) "anchor coating" where a dense, less penetrable support is first coated with another non-catalytic, more penetrable substance to provide a high surface area receptive to further impregnation by catalytic components.

Coating supports with "catalytic paint" is analogous to the method of pill coating employed by the pharmaceutical industry. Cores of support material are placed in a rotating drum and a "paint" slurry is added to coat the cores. The thickness of the catalyst coating is determined by the amount of "paint" added. A serious disadvantage of this method is that the catalyst material may peel from the support (technically termed "spall") resulting in (i) a catalytically inert support and (ii) spalling powder which will likely travel and gather downstream of the catalyst bed to distort or plug the gas flow.

Impregnation methods generally include suspending the support in a solution of the catalytic material and slowly precipitating the catalytic material onto the support or impregnating the support with the precipitant and then using a technique to force precipitation of the catalytic components immediately on the surface. Three major disadvantages are associated with impregnation methods. First, impregnation via precipitating agents may leave unwanted residues. These residues can decompose to form undesirable gases in levels unacceptable for air purification applications. Second, the catalyst precursor materials used often contain catalyst poisons, e.g. chloride, which limit the activity and effectiveness of the catalyst. Third, impregnation - and "anchor coating" and washcoating - often rely on high temperature firing or calcination to complete the coating process. Exposure to high temperatures will reduce the surface area and lower the activity of the resultant catalyst. In addition, impregnation methods involve extra steps which increases the cost of production.

Catalysts may also be prepared in powder form. Unsupported catalysts suffer from dusting, which is particularly vexing for high throughput applications. In addition, they provide poor dispersion of catalytic materials which both reduces the effectiveness and increases the cost of the catalyst for a given application.

There is an ever present need for new, improved processes for the preparation

of effective oxidation catalyst compositions. Improvements in the process which increase the versatility, effectiveness, durability, activity, and/or operating life of the oxidation catalyst satisfy this need. There is a long-standing need for an improved, efficient process of coating substrates with catalytic materials.

Summary of the Invention

The present invention is a process for forming catalysts by coating substrates with one or more catalytic components and comprises the following sequence of steps. First, the substrate is infused with an excess of solution having a starting material comprising a catalytic component precursor, where the thermal decomposition product of the catalytic component precursor is a catalytic component. Second, the excess of the solution is removed from the substrate, thereby leaving a coating of the catalytic component precursor on the surface of the substrate. Third, the coating of the catalytic component precursor is converted to the catalytic component by thermal decomposition. This is achieved by heating the coated substance to approximately 300 degrees Celsius, where the rate of heating is controlled to prevent high temperature calcination of the catalytic component precursor and where any remaining starting material is thermally decomposed, oxidized, and/or volatilized without high temperature calcining, thereby leaving only the catalytic component and no residues on the substrate surface. Finally, the coated substance is etched to increase the surface area. This can be achieved by submerging and refluxing the coated substance in a dilute solution of nitric acid.

The first three steps of the above process are repeated in order to successively layer more than one catalytic component on the substrate. In addition, the first step may be improved by infusing the substrate with an excess of solution by vacuum deaeration, and the second step may be improved by removing the excess of the solution by draining away and/or evaporating off the excess of the solution. If the catalyst is "cured" by heating in an atmosphere of a reducing gas prior to its use, its activity may be substantially improved even further. The potential reducing gases

include, but are not limited to, carbon monoxide and hydrogen.

Beneficial results are achieved where the process is used to form a catalyst consisting of a substrate and three catalytic components; wherein the substrate is selected from the group comprising ceramics, glass, metals, and fabrics; wherein the ceramic substrate is selected from the group comprising beads, pellets, and monoliths; wherein the first catalytic component is a metal oxide selected from the group comprising manganese oxide, copper oxide, and tin oxide; wherein the second catalytic component is a metal oxide, where this metal oxide is used as a promoter to increase the activity or catalyzing rate of the catalyst, and is selected from the group comprising the oxides of vanadium, chromium, copper, cobalt, manganese, nickel, or iron; and wherein the third catalytic component is a noble metal selected from the group comprising platinum, palladium, rhodium, iridium, ruthenium, osmium, and gold. Especially beneficial results are achieved where the process is used to form a catalyst consisting of a honeycomb monolith ceramic substrate successively coated with tin oxide, iron oxide, and platinum.

Drawing

Fig. 1 is a graph representing the effect of HNO_3 etching after SnO_2 loading on the conversion efficiency of Pt/ SnO_2 for CO oxidation

Example

SnEH was used to coat all the catalyst samples presented. SnEH is a liquid at room temperature. Acetone can be used to reduce the viscosity and ultimately provide thinner layers or perhaps micro-droplets of SnEH on the surface of the substrate once the acetone has evaporated. Each substrate was vacuum deaerated in SnEH either pure or diluted with acetone. Deaeration facilitates exchange of trapped air in the pores of the monolith with the desired solution insuring a thorough coating with the starting material. After deaearation, the excess solution was shaken, drained or blown from the substrate. Next the SnEH-coated substrate was placed in a muffle furnace and

heated from ambient temperature to 200°C at 1°C/min and held for 3-6 hours and then heated to 300°C at 1°C/min for 2-6 hours to decompose the SnEH to tin oxide and remove residual organic material, respectively. The above steps were repeated until the desired loading of tin oxide was obtained. The SnO_2- coated substrate was etched to increase the surface area of the SnO_2 layer which ultimately provides better dispersion of the noble metal. The SnO_2 -coated substrates were submerged in 1.5 to 1.6 M nitric acid and heated to near boiling. Etching time varies depending on the desired surface area of the SnO_2 -coated substrate. Once etched, the substrates were rinsed with hot DI water to remove any residual nitric acid. Finally the substrates were placed into a muffle furnace and dried at 100°C for 1 hour then to 250°C for 2 hours to decompose any nitric acid. CO oxidation activity improved 300-400% for the same amount of noble metal when compared to non-etched substrates or substrates etched prior to the application of the first catalytic component.

The present invention has been described in detail with respect to certain preferred embodiments thereof. However, as is understood by those of skill in the art, variations and modifications in this detail can be made without any departure from the spirit and scope of the present invention as defined in the hereto-appended claims.

Claims

1. A process of coating the surface of a substrate with catalytic components to form a catalyst, where the catalyst is a catalyst matrix having two or more catalytic components which are layered successively on the substrate, comprising the following sequence of steps:

(a) infusing the substrate with more than an adequate amount of solution having a starting material comprising a catalytic component precursor, where the thermal decomposition product of the catalytic component precursor is a catalytic component and where an adequate amount of solution is an amount that thoroughly coats the substrate;

(b) removing from the substrate any solution in excess of an adequate amount, thereby leaving a coating of the catalytic component precursor on the surface of the substrate;

(c) heating the coated substrate thereby converting the coating of the catalytic component precursor to the catalytic component by thermal decomposition,

(d) etching the coated substrate,

(e) repeating steps (a) - (c) for a second catalytic component.

2. The process of claim 1, wherein the sequence of steps (a), (b), and (c) is repeated in order to successively layer more than two catalytic components on the substrate.

3. The process of claim 1, wherein the substrate is a substrate selected from the group consisting of ceramics, glass, metals, and fabrics.

4. The process of claim 3, wherein the substrate is a ceramic substrate selected from the group consisting of beads, pellets, and monoliths.

5. The process of claim 4, wherein the ceramic substrate is a monolith.

6. The process of claim 1, wherein a first catalytic component is a metal oxide.

7. The process of claim 6, wherein the first catalytic component is a metal oxide selected from the group consisting of manganese oxide and tin oxide.

8. The process of claim 7, wherein the first catalytic component is tin oxide.

9. The process of claim 1, wherein a second catalytic component is a noble metal.

10. The process of claim 9, wherein the second catalytic component is a noble metal selected from the group consisting of platinum and palladium.

11. The process of claim 10, wherein the second catalytic component is platinum.

12. The process of claim 2, wherein a third catalytic component is a metal oxide and this metal oxide is used as a promoter, where the promoter is a catalytic component which increases the activity or catalyzing rate of the catalyst.

13. The process of claim 12, wherein the third catalytic component is a metal oxide selected from the group consisting of the oxides of manganese and iron.

14. The process of claim 13, wherein the third catalytic component is iron oxide.

15. The process of claim 2, wherein the catalyst has three catalytic components.

16. The process of claim 15, wherein the three catalytic components are a first metal oxide, a second metal oxide, and a noble metal.

17. The process of claim 16, wherein the first metal oxide is tin oxide, the second metal oxide is iron oxide, and the noble metal is platinum.

18. The process of claim 1, wherein the substrate, now coated with one or more catalytic components, is heated in an atmosphere containing a reducing gas.

19. The process of claim 18, wherein the reducing gas is either carbon monoxide or hydrogen.

20. The process of claim 1, wherein step (a) is modified so that the substrate is infused with an excess of the solution by vacuum deaeration.

21. The process of claim 1, wherein step (b) is modified so that the excess of the solution is removed by draining away and/or evaporating off the excess of the solution.

22. The process of claim 1, wherein step (c) is modified so that the heating of the coated substrate is to approximately 300 degrees Celsius.

23. The process of claim 1, wherein said process is used for the oxidation of carbon monoxide.

24. The process of claim 1, wherein said process is used for the oxidation of volatile organic compounds.

PROCESS FOR COATING SUBSTRATES WITH CATALYTIC MATERIALS

Abstract

The present invention is a process for forming catalysts by coating substrates with two or more catalytic components and comprises the following sequence of steps. First, the substrate is infused with an excess of solution having a starting material comprising a catalytic component precursor, where the thermal decomposition product of the catalytic component precursor is a catalytic component. Second, the excess of the solution is removed from the substrate, thereby leaving a coating of the catalytic component precursor on the surface of the substrate. Third, the coating of the catalytic component precursor is converted to the catalytic component by thermal decomposition. Finally, the coated substance is etched to increase the surface area. The first three steps are then repeated for at least a second catalytic component. This process is ideally suited for application in producing efficient low temperature oxidation catalysts.

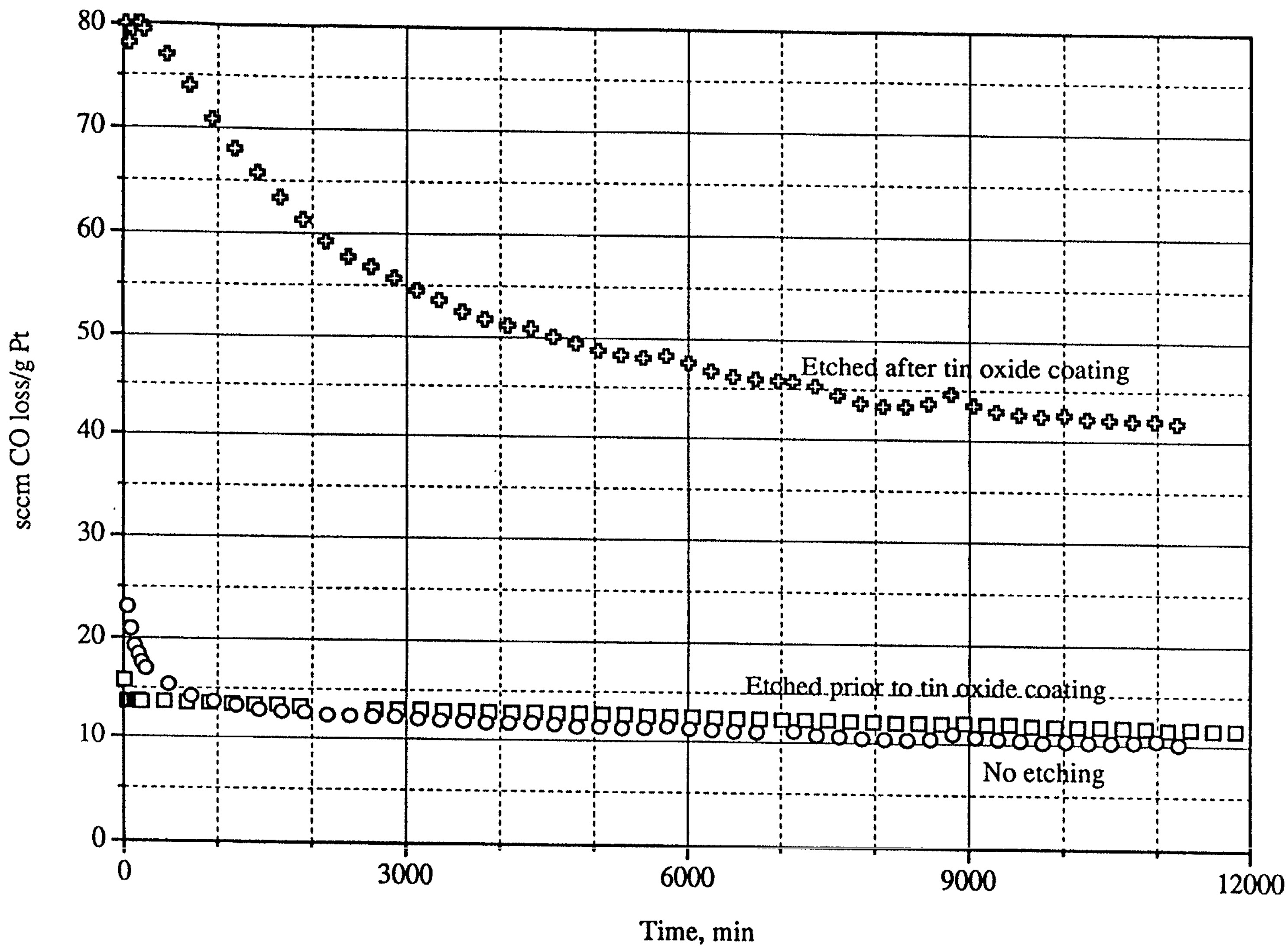

FIG. 1

0960721 .063000

EK 456804778

National Aeronautics and
Space Administration

NASA Case No.: 15851-1

DECLARATION, POWER OF ATTORNEY AND PETITION - ORIGINAL APPLICATION

As a below named inventor, I hereby declare that: my residence, post office address and citizenship, are stated below next to my name, I believe I am the original, first and sole inventor (If only one name Is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled PROCESS FOR COATING SUBSTRATES WITH CATALYTIC MATERIALS , the specification of which √ is attached hereto, ___was filed on (Date) ________________ as Application Serial No. __________ and was amended on (Date)____________________.

I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose to the Patent and Trademark Office all information which is known to me to be material to patentability as defined in 37 CFR §1.56.

I hereby claim the benefit under 35 U.S.C. §120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of 35 U.S.C. §112, I acknowledge the duty to disclose to the Patent and Trademark Office all information known to me to be material to patentability as defined in 37 CFR §1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application:_____ _ (Serial No.), __________ (Filing Date) ,the status of which is

_ patented, _ pending, _ abandoned.

I hereby claim foreign priority benefits under Title 35, United States Code, § 119(e) of any United States Provisional applications listed below:

PROVISIONAL APPLICATION NUMBER	FILING DATE
60 / 141,729	JUNE 30, 1999
____ /	

the status of which is

_ patented, X pending, _ abandoned.

POWER OF ATTORNEY: I hereby appoint the following attorney(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

LINDA B. B. BLACKBURN	Reg. No. 38,385
KURT G. HAMMERLE	Reg. No. 36,819
ROBIN W. EDWARDS	Reg. No. 39,179
HILLARY W. HAWKINS	Reg. No. 42,235
HELEN M. GALUS	Reg. No. 40,615
SUE H. PALK	Reg. No. 36,422
GARY G. BORDA	Reg. No. 35,455
ALAN J. KENNEDY	Reg. No. 28,625
HARRY LUPULOFF	Reg. No. 31,117

ADDRESS ALL CORRESPONDENCE TO:

Name: HILLARY W. HAWKINS

Address: NASA LANGLEY RESEARCH CENTER
MAIL STOP 212
3 LANGLEY BOULEVARD
HAMPTON, VA 23681-2199

DIRECT TELEPHONE CALLS TO:

Name: HILLARY W. HAWKINS

Telephone: (Set out complete number to be dialed from USPTO):
757-864-8882
757-864-3522

Further, as a named inventor I certify that the Government of the United States of America, as represented by the Administrator of the National Aeronautics and Space Administration, has _x_ an assignment in, or _ license to the invention set forth in this application and has the irrevocable right to practice this application and to receive the patent.

Wherefore, I pray that Letters Patent be granted to me for this invention or discovery described and claimed in the foregoing specification and claims, and I hereby subscribe my name to the foregoing specification, claims, power of attorney and this petition.

I hereby declare that all statements made herein of my own knowledge are true and that - statements made on information and belief are believed to be true; and further that these, statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. §1001; and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

FULL NAME OF INVENTOR	LAST UPCHURCH	FIRST Billy	MIDDLE OR INITIAL T.
RESIDENCE AND CITIZENSHIP	CITY Virginia Beach	STATE OR FOREIGN COUNTRY Virginia	COUNTRY OF CITIZENSHIP U.S.
POST OFFICE	STREET NO. AND NAME 1213 Bluebird Drive	CITY AND STATE OR (COUNTRY) Virginia Beach, Virginia	ZIP CODE 23451
SIGNATURE			DATE

FULL NAME OF INVENTOR	LAST SCHRYER	FIRST David	MIDDLE OR INITIAL R.
RESIDENCE AND CITIZENSHIP	CITY Hampton	STATE OR FOREIGN COUNTRY Virginia	COUNTRY OF CITIZENSHIP U.S.
POST OFFICE	STREET NO. AND NAME 106 Santa Barbara Drive	CITY AND STATE OR (COUNTRY) Hampton, Virginia	ZIP CODE 23666
SIGNATURE			DATE

FULL NAME OF INVENTOR	LAST KIELIN	FIRST Eric	MIDDLE OR INITIAL J.
RESIDENCE AND CITIZENSHIP	CITY Alexandria	STATE OR FOREIGN COUNTRY Virginia	COUNTRY OF CITIZENSHIP U.S.
POST OFFICE	STREET NO. AND NAME 6374 Beryl Road, Apt. 203	CITY AND STATE OR (COUNTRY) Alexandria, Virginia	ZIP CODE 22312
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